Designation: D3186 – 07 (Reapproved 2016)

Standard Test Methods for Rubber—Evaluation of SBR (Styrene-Butadiene Rubber) Mixed With Carbon Black or Carbon Black and Oil[1]

1. Scope

1.1 These test methods cover the standard materials, test formula, mixing procedures, and test methods for the evaluation and production control of pigmented types of styrene-butadiene rubbers (SBR). This includes the pigmented SBR oil masterbatches.

1.2 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D412 Test Methods for Vulcanized Rubber and Thermoplastic Elastomers—Tension
D1646 Test Methods for Rubber—Viscosity, Stress Relaxation, and Pre-Vulcanization Characteristics (Mooney Viscometer)
D2084 Test Method for Rubber Property—Vulcanization Using Oscillating Disk Cure Meter
D3182 Practice for Rubber—Materials, Equipment, and Procedures for Mixing Standard Compounds and Preparing Standard Vulcanized Sheets
D3896 Practice for Rubber From Synthetic Sources—Sampling
D4483 Practice for Evaluating Precision for Test Method Standards in the Rubber and Carbon Black Manufacturing Industries
D5289 Test Method for Rubber Property—Vulcanization Using Rotorless Cure Meters
D6204 Test Method for Rubber—Measurement of Unvulcanized Rheological Properties Using Rotorless Shear Rheometers

3. Significance and Use

3.1 These test methods are intended mainly for referee purposes but may be used for quality control of masterbatch production. They may also be used in research and development work and for comparison of different rubber samples in a standard formula.

3.2 These test methods may also be used to obtain values for customer acceptance of rubber.

4. Standard Test Formula

4.1 *Standard Formula*—See Table 1.

5. Sample Preparation

5.1 Obtain and prepare the test samples in accordance with Practice D3896.

6. Mixing Procedures

6.1 The compound may be prepared either on a mill, miniature internal mixer, or laboratory internal mixer, although slightly different results may be obtained.

6.2 *Mill Mix Procedure:*

6.2.1 For general mixing procedures, refer to Practice D3182.

6.2.2 *Mill Mixing Cycle*—See Table 2.

6.2.2.1 After mixing according to Table 2, measure and record the batch mass. If it differs from the theoretical value by more than 0.5 %, discard the batch.

6.2.2.2 If required, cut samples from the batch to allow testing of compound viscosity and processability in accordance with Test Methods D1646 or D6204, and vulcanization characteristics in accordance with Test Methods D2084 or D5289.

6.2.2.3 If tensile stress strain tests are required, sheet off to a finished thickness of approximately 2.2 mm (0.087 in.) and condition the compound according to Practice D3182.

6.3 *Miniature Internal Mixer (MIM) Procedure:*

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[1] These test methods are under the jurisdiction of ASTM Committee D11 on Rubber and are the direct responsibility of Subcommittee D11.23 on Synthetic Rubbers.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

TABLE 1 Standard Pigmented SBR Test Formula

Material	SRM/IRM No.[A]	Quantity, Parts by Mass
Masterbatch	...	100 + X[B] + Y[C]
Zinc oxide[D,E]	[A]	3.00
Sulfur[D,E]	[A]	1.75
Stearic acid[D, E]	[A]	1.50
TBBS[E,F]	[A]	1.25
Total		107.50 + X + Y
Batch factor[G]		

[A] Use current IRM/SRM.

[B] X = parts carbon black per 100 parts base polymer.

[C] Y = parts oil per 100 parts base polymer.

[D] For the MIM procedure, it is recommended that a blend of compounding materials be prepared to improve accuracy of the weighing of these materials. This material blend is prepared by blending a proportional mass of each material in a dry powder blender such as a biconical blender or vee blender. A mortar and pestle may be used for blending small quantities.

[E] For mill mixes, weigh the rubber to the nearest 1.0 g, the sulfur and the accelerator to the nearest 0.02 g, and all of the other compounding materials to the nearest 0.1 g. For MIM mixes, weigh the rubber and material blend to the nearest 0.01 g and individual pigments, if used, to the nearest 0.001 g.

[F] TBBS is *N-tert*-butyl-2-benzothiazolesulfenamide.

[G] For mill mixes, a batch factor should be selected to the nearest 0.5 to give as large a total mass as possible that will not exceed 525.0 g. Calculate all parts to the nearest 0.01 part. For MIM mixes, calculate a batch factor to the nearest 0.01 that will provide a 75 % loading of the mixing chamber.

TABLE 2 Mill Mixing Cycle

	Duration, min	Accumulative, min
Set the mill opening at 1.40 mm (0.056 in.) and band the masterbatch on the slow roll without cutting.	0	0
Add sulfur slowly and evenly across the mill at a uniform rate.	2	2
Add stearic acid. Make one ¾ cut from each side after the stearic acid has been incorporated.	2	4
Add zinc oxide and TBBS accelerator.	3	7
Make three ¾ cuts from each side and cut the batch from mill.	2	9
Set the rolls at 0.8 mm (0.032 in.). Pass the rolled stock endwise through the mill six times.	2	11
Open the mill to give a minimum stock thickness of 6 mm (0.25 in.) and pass the compound through the mill four times, folding it back on itself each time.	1	12

6.3.1 For general mixing procedures refer to Practice D3182.

6.3.2 *MIM Mixing Cycle*—See Table 3.

6.3.2.1 After mixing according to Table 3, turn off the motor, raise the ram, remove the mixing chamber and discharge the batch. Record the maximum batch temperature indicated, if desired.

6.3.2.2 Immediately pass the discharge from the mixer twice through a standard mill maintained at 50 ± 5°C (122 ± 9°F) with a roll separation of 0.5 mm (0.020 in.) once, then twice at a separation of 3 mm (0.12 in.) in order to dissipate heat. Pass the rolled batch endwise through the mill six times with an opening of 0.8 mm (0.032 in.) to enhance the dispersion.

TABLE 3 Miniature Internal Mixer Cycle

	Duration, min	Accumulative, min
Charge the mixing chamber with the masterbatch strips, lower the ram, and start the timer.	0.0	0.0
Masticate the masterbatch.	0.5	0.5
Raise the ram, and add previously blended zinc oxide, sulfur, stearic acid, and TBBS, taking care to avoid any loss. Sweep the orifice and lower the ram.	0.5	1.0
Allow the batch to mix.	8.0	9.0

6.3.2.3 Measure and record the batch mass. If it differs from the theoretical value by more than 0.5 %, discard the batch.

6.3.2.4 If required, cut samples from the batch to allow testing of compound viscosity and processability in accordance with Test Methods D1646 or D6204, and vulcanization characteristics in accordance with Test Methods D2084 or D5289.

6.3.2.5 If tensile stress strain tests are required, sheet off to a finished thickness of approximately 2.2 mm (0.087 in.) and condition the compound according to Practice D3182.

6.4 *Internal Mixer Procedure:*

6.4.1 For general mixing procedure refer to Practice D3182.

6.4.2 *Mixing Cycle-Initial Mix*—See Table 4.

6.4.2.1 After mixing according to Table 4, measure and record the batch mass. If it differs from the theoretical value by more than 0.5 %, discard the batch.

6.4.2.2 Pass the batch immediately through the standard laboratory mill three times, with a mill opening of 6.0 mm (0.25 in.) and roll temperature of 40 ± 5°C (104 ± 9°F).

6.4.2.3 Allow the batch to rest for 1 to 24 h.

6.4.3 *Final Mix*—See Table 5.

6.4.3.1 After mixing according to Table 5, measure and record the batch mass. If it differs from the theoretical value by more than 0.5 %, discard the batch.

6.4.3.2 If required, cut samples from the batch to allow testing of compound viscosity and processability in accordance with Test Methods D1646 or D6204, and vulcanization characteristics in accordance with Test Methods D2084 or D5289.

6.4.3.3 If tensile stress strain tests are required, sheet off to a finished thickness of approximately 2.2 mm (0.087 in.) and condition the compound according to Practice D3182.

7. Preparation and Testing of Vulcanizates

7.1 For stress-strain testing, prepare the test sheets and vulcanize them in accordance with Practice D3182.

7.1.1 The recommended standard cure times for the mill mixes are 25, 35, and 50 min at 145°C (293°F). The recommended cure time for the miniature internal mixer and the internal mixer compounds is 35 min at 145°C (293°F).

7.1.2 Condition the cured sheets for 16 to 96 h at a temperature of 23 ± 2°C (73.4 ± 3.6°F).

NOTE 1—Quality control of rubber production may require testing within 1 to 6 h to provide close surveillance; however, slightly different results may be obtained.

TABLE 4 Internal Mixer - Initial Cycle

	Duration, min	Accumulative, min
Adjust the internal mixer temperature to achieve the discharge conditions outlined below. Close the discharge gate, start the rotors at 8.1 rad/s (77 r/min), and raise the ram.	0.0	0.0
Charge one-half of the rubber, all of the zinc oxide, stearic acid, and then the other one-half of the rubber. Lower the ram.	0.5	0.5
Allow the batch to mix.	3.0	3.5
Raise the ram, and clean the mixer throat and the top of the ram. Lower the ram.	0.5	4.0
Allow the batch to mix until a temperature of 170°C (338°F) or a total of 6 min is reached, whichever occurs first. Discharge the batch.	2.0	6.0

TABLE 5 Internal Mixer - Final Cycle

	Duration, min	Accumulative, min
Adjust the internal mixer temperature to 40 ± 5°C (104 ± 9°F), turn off steam and turn on full cooling water to the rotors, start the rotors at 8.1 rad/s (77 r/min), and raise the ram.	0.0	0.0
Charge one-half of the batch, with all the sulfur and accelerator rolled into this portion of the batch before feeding to the mixer. Add the remaining portion of the batch. Lower the ram.	0.5	0.5
Allow the batch to mix until a temperature of 110 ± 5°C (230 ± 9°F) or a total mixing time of 3 min is reached, whichever occurs first. Discharge the batch.	2.5	3.0
With the rolls of a standard laboratory mill maintained at 40 ± 5°C (104 ± 9°F), and set at 0.8 mm (0.032 in.) opening, pass the rolled batch endwise through the rolls six times.	2.0	5.0
Open the rolls to give a minimum thickness of 6 mm (0.25 in.) and pass the compound through four times, folding it back on itself each time.	1.0	6.0

7.1.3 Prepare test specimens and obtain modulus, tensile and elongation in accordance with Test Methods D412.

7.2 An alternative to measuring vulcanization characteristics by means of tensile stress measurement on vulcanizates is the measurement of vulcanization characteristics in accordance with Test Method D2084 (Oscillating Disk Cure Meter Method) or Test Method D5289 (Rotorless Cure Meter Method). These methods will not produce equal results.

7.2.1 The recommended Test Method D2084 test conditions are 1.67 Hz (100 cpm) oscillation frequency, 1° oscillation amplitude, 160°C die temperature, 30-min test time, and no preheating. The recommended Test Method D5289 test conditions are 1.67 Hz (100 cpm) oscillation frequency, 0.5° oscillation amplitude, 160°C die temperature, 30-min test time, and no preheating. Test condition tolerances are specified by the test methods.

7.2.2 The recommended standard test parameters are M_L, M_H, t_{s1}, t'50, and t'90.

NOTE 2—It is recommended that M_H, if applicable, be taken as the torque value at 30 min.

8. Precision and Bias

8.1 This precision and bias section has been prepared in accordance with Practice D4483. Refer to Practice D4483 for terminology and other statistical calculation details.

8.2 The precision results in this precision and bias section give an estimate of the precision of these test methods with the materials (rubbers) used in the particular interlaboratory program as described below. The precision parameters should not be used for acceptance/rejection testing of any group of materials without documentation that they are applicable to those particular materials and the specific testing protocols that include these test methods.

8.3 A Type 2 (interlaboratory) precision was evaluated. Both repeatability and reproducibility are short term; a period of a few days separates replicate test results. A test result is the average value, as specified by these test methods, obtained on one determination(s) or measurement(s).

8.4 Two different materials (SBR rubbers) were used in the interlaboratory program; were tested in six laboratories on two different days, by the internal mixer method. The results of the precision calculations for repeatability and reproducibility are given in Table 6, as conducted according to Test Method D2084.

8.5 The precision of these test methods may be expressed in the format of the following statements that use an "appropriate value" of r, R, (r), or (R), to be used in decisions about test results. The appropriate value is that value of r or R associated with a mean level in Table 6 closest to the mean level under consideration at any given time for any given test parameter, and for any given material in routine testing operations.

8.6 *Repeatability*—The repeatability, r, of these test methods has been established as the appropriate value tabulated in Table 6. Two single test results, obtained under normal test method procedures, that differ by more than this tabulated r (for any given level) must be considered as derived from different or nonidentical sample populations.

8.7 *Reproducibility*—The reproducibility, R, of these test methods has been established as the appropriate value tabulated in Table 6. Two single test results obtained in two different laboratories, under normal test method procedures, that differ by more than the tabulated R (for any given level) must be considered to have come from different or nonidentical sample populations.

8.8 Repeatability and reproducibility expressed as a percentage of the mean level, (r) and (R), have equivalent application statements as above for r and R. For the (r) and (R) statements, the difference in the two single test results is expressed as a percentage of the arithmetic mean of the two test results.

8.9 *Bias*—In test method terminology, bias is the difference between an average test value and the reference (or true) test

TABLE 6 Type 2 Precision for Curemeter Parameters

NOTE 1—
Sr = repeatability standard deviation, in measurement units,
r = repeatability, in measurement units,
(r) = repeatability, (relative) percent,
SR = reproducibility standard deviation, in measurement units,
R = reproducibility, in measurement units, and
(R) = reproducibility, (relative) percent.

Property	Units	Range of Values[A]	Within Laboratory			Between Laboratories		
			Sr	*r*	(*r*)	*SR*	*R*	(*R*)
M_L	dN·m	6.4 to 8.2	0.17	0.48	6.6	0.92	2.60	35.6
M_H	dN·m	24.2 to 42.7	0.69	1.95	5.8	2.69	7.61	22.7
t_{s1}	min	5.8 to 6.8	0.19	0.54	8.6	0.89	2.52	40.0
t' 50	min	9.3 to 9.9	0.28	0.79	8.2	0.73	2.07	21.6
t' 90	min	15.0 to 15.1	0.40	1.13	7.5	0.86	2.43	16.2

[A] Measured at 160°C, 1.7 Hz, 1° amplitude arc—midpoint of range used for (*r*) and (*R*) calculations.

property value. Reference values do not exist for these test methods, since the value (of the test property) is exclusively defined by the test methods. Bias therefore cannot be determined.

9. Keywords

9.1 carbon black and oil; mixed with carbon black; SBR

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